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Development of the Test Kit for Fuel Acid Contamination

February 1995

By Shing-Bong Chen
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USA Tank Automotive Command
Mobility Technology Center Belvoir

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13. ABSTRACT *(Maximum 200 words)* This report describes efforts to develop a test method for determining the presence of a fuel acid contaminant used as a possible anti-materiel agent in military fuels. The test method must be usable in a field environment. After investigation of the properties of candidate agents and evaluation of a substitution reaction detection method, the use of pH indicator solutions following water extraction was determined to be the most appropriate for this application. The report also describes the fabrication of test models of the Test Kit for Fuel Acid Contamination for use by troops in the latter stages of *Operation Desert Storm.* A copy of a draft Purchase Description that can be used for possible future procurement is included.

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Fuels and Lubricants Division

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Section 1 Introduction

OBJECTIVE

This report covers efforts directed towards the development of the Test Kit for Fuel Acid Contamination (TKFAC).

BACKGROUND

Intelligence reports generated in 4QFY90 and 1QFY91 during Operation Desert Shield indicated a possible threat to U.S. military fuel stocks in Southwest Asia. Specially, a fuel acid anti-materiel contamination agent added to hydrocarbon fuels could possibly damage engines, fuel control systems and fuels handling equipment. Consequently, a need was expressed by the intelligence community and other DA activities for a test kit to detect the presence of this "acid contaminant" in the field. The test kit would be used by field soldiers or petroleum handling personnel at wholesale fuel distribution points. Based on this expressed need, a research and development effort was initiated by the then Army Belvoir Research Development and Engineering Center (now the Army Tank Automotive Research, Development and Engineering Center, Mobility Technology Center-Belvoir).

Section 2 Investigation

APPROACH

The purpose of this effort was to develop an expedient test procedure, applicable to a field test kit, capable of detecting the presence of a specific acid contamination agent in hydrocarbon fuels. Tests were to be simple and reliable procedures based on proven analyses techniques. One or more prototypes would be fabricated based on the findings of the test program. The resulting test kit would require a minimum of new components, be man-portable and capable of being utilized by field soldiers or petroleum handling personnel in a field environment.

Phosphorous-halides and their organic derivatives are typical acid contaminant agents in hydrocarbon fuels. These compounds will react with water, alcohols and amines to yield hydrogen chloride (HCl) and phosphorous reaction products. Other non-phosphorous acid contaminants can also react with water producing HCl. Hydrogen chloride in an aqueous solution produces hydrochloric acid which can attack metallic surfaces, particularly ferrous metals, copper and aluminum, in engines and fuels handling equipment. Water is almost always present in fuel (suspended or lying on the bottom of tanks) and always present as a combustion product.

The research effort focused on the conversion of the acid contaminant Agent in fuels to hydrochloric acid with subsequent detection of the acid. Other reaction products which may be potentially harmful to consuming equipment and may be fuel soluble may or may not be detectable. Two detection methods were investigated.

Water extraction followed by detection by acid-base (pH) indicator. Test fuels were treated with acid contaminant Agents at specified levels. The fuels were contacted (by agitation) with water containing a range of pH indicator fluids. The aqueous layer was allowed to separate and examined for color. The color of the aqueous layer indicated the acidity (pH level) of the extract. A pH level substantially lower than that of a blank sample, i.e., neat fuel without contaminant, indicated that the test fuel had been contaminated.

Water extraction followed by detection by silver chloride precipitation. Test fuels treated with acid contaminant agents at specified levels were contacted with an aqueous silver nitrate solution. The aqueous layer was allowed to separate and examined for the presence of a silver chloride precipitate. Silver chloride has very low solubility in water and the present of the precipitate would indicate the presence of a chloride type contamination in the fuel.

EXPERIMENTAL

Test Materials

Acid contaminant agents. Five commercially available chemicals (coded as Agents A, B, C, D, and E), representative of known acid contaminant agents, were used in all tests. The chemicals were introduced into the test fuels at a concentration of 100 parts per million (ppm). This concentration was selected based on information that this is the minimum level required to be effective as an acid contaminant in fuel.

Test Fuels. Military and commercial fuels were used as follows:

Turbine fuels:
Jet A per ASTM D 1655
JP-5 per MIL-T-5624
JP-8 per MIL-T-83133

Diesel fuels:
DF-2 per VV-F-800
Referee grade diesel fuel per MIL-F-46162, Type I
Reference diesel Caterpillar fuel (CAT-1H2)

Gasolines:
Regular grade unleaded gasoline per ASTM D 4814
Leaded gasoline (AVGAS) per ASTM D 910

Indicator solutions. Commercially available Methyl Orange, and Bromocresol Green (0.04% solution) were used to prepare pH indicator solution for the acid-base indicator tests.

Commercially available silver nitrate solution, 1.0 N, was used for the silver chloride precipitation test.

Instrumentation

Acid levels (pH values) were determined by a Mettler model DL25 titrator using a DG calomel reference electrode and a glass electrode.

Nuclear Magnetic Resonance (NMR) spectra were taken to indicate the presence of the agents using a Varian VXR-300 NMR spectrometer. The spectrometer was operated at 75.424 MHz with deuteriated chloroform (CDCl3) used to provide an internal lock signal and as a reference for carbon-13 NMR. The spectrometer was operated at 121.413 MHz without lock signal and triphenyl phosphate was used as an external reference for phosphorous-31 NMR.

Procedure

Preparation of doped fuels. One drop (approximately 7 mg) of each acid contamination agent was placed in a beaker then diluted with test fuel to make a solution of 100 ppm agent by weight. Fuel solutions were produced for each of the test fuels and stored in glass bottles until used.

Water extraction of doped fuels. Ten (10) mL quantities of doped test fuels or neat fuels (used as a blank) were contacted with ten (10) mL of water containing the detection agents listed above to extract the acid contaminant. The resulting aqueous layer was then examined for color change or the presence of a silver chloride precipitate.

RESULTS AND DISCUSSION

Selection of pH indicating solution. The acid contamination agents react with water quantitatively to yield one mole of acid per mole of water. A level of 100 ppm of agent in pH "neutral" fuel will react and be extracted by an equal quantity of water to give an aqueous layer having a pH value between 3.0 and 3.2. Certain fuels containing one mole of agent could react to yield more than one mole of acid possibly resulting in a pH level below 3.0.

Methyl Orange solution is commonly used as an indicator in acid/base titration. The pH interval and corresponding color changes for Methyl Orange is between 3.1 (red) and 4.4 (yellow-orange), which is close to the detection limits for 100 ppm acid contamination. In order to enhance the color change at the 100 ppm level, another indicator, Bromocresol Green, was blended with the Methyl Orange. A mixture of 0.008% Methyl Orange and 0.002% Bromocresol Green was found to be a highly satisfactory indicator solution for the purpose of this project. The mixture exhibits a dark green-blue at pH 6.2, changes to light green at pH 4.0, yellow orange at pH 3.5, and orange at pH 3.0 as the solution increases in acidity. Thus, a color change from green-blue or green to yellow or yellow orange is a positive indication of acid contaminant in the fuel.

Extreme temperature tests were performed on the indicating solution mixture to ensure its stability. The indicating solution was kept at minus 20°C for 72 hours, resulting in freezing, and then allowed to thaw. The same mixture was kept at plus 65°C for 30 hours. The indicating solution did not show any degradation as a result of the extreme temperature exposures.

Stability of doped fuels. Five acid contaminant agents, liquids at room temperature, were added to test fuels. All agents appeared to be fully solubilized. Agents A, B and E were added to all test fuels. Agents C and D were only added to Jet A and referee grade diesel fuel. Some visual observations are as follows:

Reference diesel Caterpillar fuel (CAT-1H2). The light yellowish fuel when doped with agent A became slightly darker after three weeks. However, the acidity of fuel remained unchanged. The CAT 1-H appears to be more stable towards acid contamination agents than other distillate fuels.

Referee grade diesel fuel. The medium brown fuel changed to a much darker brown after doping with the agents. A large quantity of dark precipitate settled to the bottom of the beaker the day after doping, compared with other fuels. Referee grade fuel is intended to be the worst case diesel fuel with high levels of sulfur and other polar compounds. The agents are known to react with alcohols, amines, sulfides and other polar substances.

Diesel fuel. The yellow fuel changed to dark brown when doped with the agents, similar to the color of neat referee grade diesel fuel.

Jet A fuel. The near water white fuel remained clear when doped with Agent A but turned slightly hazy and developed a purple hue by the next day. Later, the purple settled out as a precipitate on the bottom and sides of the beaker. The purple material could have been the result of the agents reacting with fuel components and producing fuel insolubles.

JP-5 and JP-8 fuels. The pale yellow fuels initially showed haziness after doping. But the haziness disappeared as a purple precipitate came out. The quantity of precipitate was larger then that obtained from Jet A. The mandatory military additives, Fuels System Icing Inhibitor (FSII), corrosion inhibitor, and Static Dissipator Additive (SDA), found in JP-5 and JP-8, but not in Jet A, may have contributed to the increased precipitate. These additives are all polar and expected to react with the Agents.

Gasoline, leaded and unleaded. These fuels, after being doped with Agent A, exhibited much less color change, haziness or precipitation then the other fuels tested.

Water extraction followed by indicator solution. All fuels doped at the 100 ppm level tested positively for acid contaminant agent when evaluated by extraction followed by pH indicating solution. The color change was generally from dark blue-green to yellow orange.

The high sulfur referee diesel fuel used in the tests was found to have a higher natural acidity than any fuel tested. However, the natural acidity was not enough to indicate a false positive.

Jet A samples doped at both the 100 and 50 ppm levels with Agent A indicated positive results. However, acid levels in these fuels tended to decrease with time. It was observed that the purple hue initially present in the fuel came out as a purple colored precipitate with the fuel becoming clear again. Presumably, the acidic compounds settled out with the precipitate. For example, a 100 ppm agent doped fuel indicated, after one week, a level equivalent to fuel freshly prepared with agent at levels between 25 and 50. The Fuel System Icing Inhibitor (FSII) seems to promote this phenomenon. A doped Jet A fuel containing a high concentration of diethylene glycol monomethyl ether (a FSII additive) did not give a noticeably different positive reading than a similarly doped Jet A sample without the FSII. However, after one week, considerably less acidity was observed, equivalent to less than 25 ppm agent doped fuel.

Jet A was doped with approximately 0.25 vol. percent of MIL-L-2104D engine motor oil before addition of Agent A. This fuel was then subjected to water extraction with pH indicating solution and indicated positive at both the 50 and 100 ppm acid levels. However, the resulting color of the water extract was lighter than those for fuels without motor oil. In addition, the fuel layer showed a blue hue. The blue could be due to the additives in oil increasing the solubility of the Bromocresol Green in the fuel shifting the hue towards blue. Motor oil frequently contains detergents which can cause interference but this would tend to produce false negative results, not false positives.

Nuclear Magnetic Resonance (NMR) investigation. The chemical shift (ppm levels) of nuclei vary with the chemical environment and are used in qualitative analysis. The location of the peaks are used for quantitative analysis.

The purple settlements from Agent A doped Jet A fuel were washed with heptane and dissolved in deuterated chloroform. The P31-NMR spectrum (Figure 1) showed that the settlement contained a high concentration of phosphorous compounds which was different from that obtained from Agent A alone in Jet A fuel (Figure 2). An equal amount of water was added to doped fuel without agitation forming two layers, the phosphorous compound (Agent A) in the fuel layer gradually diminished to undetectable levels by the following day while the water layer seemed to produce another phosphorous compound (Figure 3) which differed from the purple material. The carbon-13 NMR revealed that the purple material was very complex with a mixture of hydrocarbons.

The water extraction of Agent C from Jet A gave another phosphorus compound shown in Figure 4. No detectable Agent C was found in the fuel layer indicating that the reaction with water was complete. All of the phosphorous reaction products seem to be more soluble in water then in fuel.

The spectra of the phosphorous Agents B, C, and D in Jet A fuel are shown as Figures 5, 6, and 7. The chemical shifts of each Agent are different and can be used to identify the type of Agent introduced into the fuel.

Water extraction with silver nitrate solution. The reaction of Agents with water was expected to produce chloride ions which can be precipitated by silver nitrate to form insoluble silver chloride. Silver chloride, being insoluble in water or fuel, would show up as a white precipitate. The silver chloride that did form occurred at the fuel-water interface and was not highly visible. This precipitate could have been easily masked by particulate matter, common in diesel fuels, that tend to float at the fuel-water interface. Consequently, this approach was abandoned in the early stages of this effort.

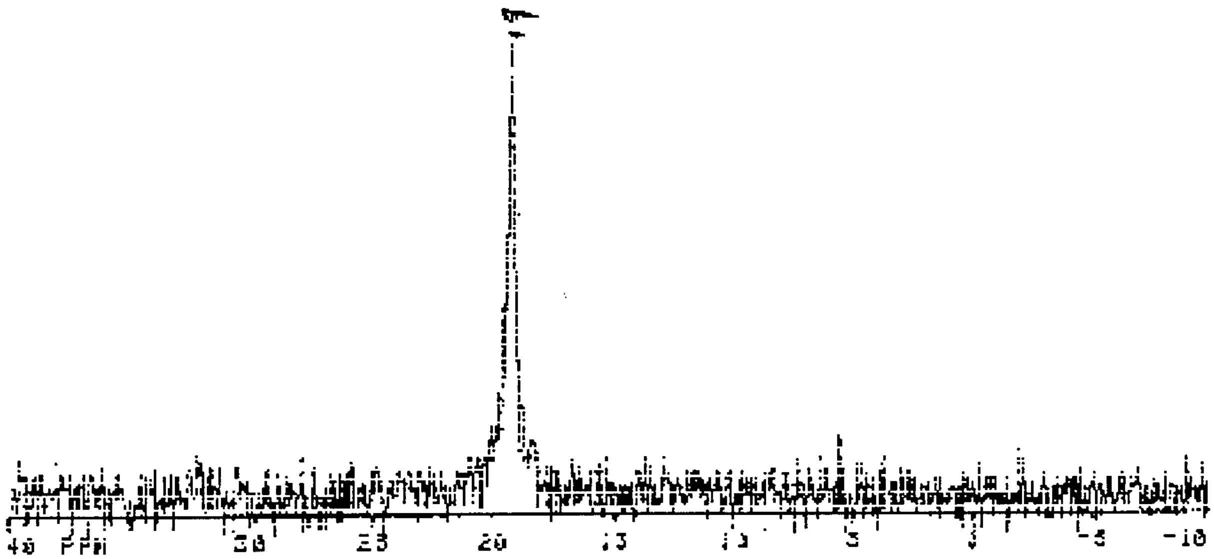

Figure 1. NMR Spectrum of Settlement from Agent A in Jet A Fuel

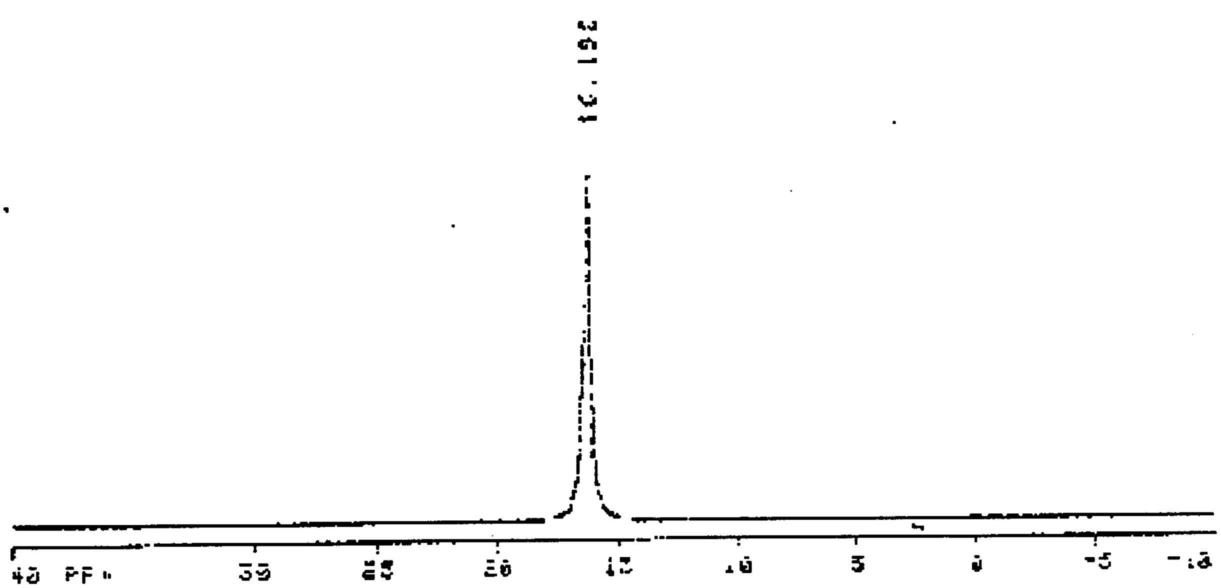

Figure 2. NMR Spectrum of Agent A in Jet A Fuel

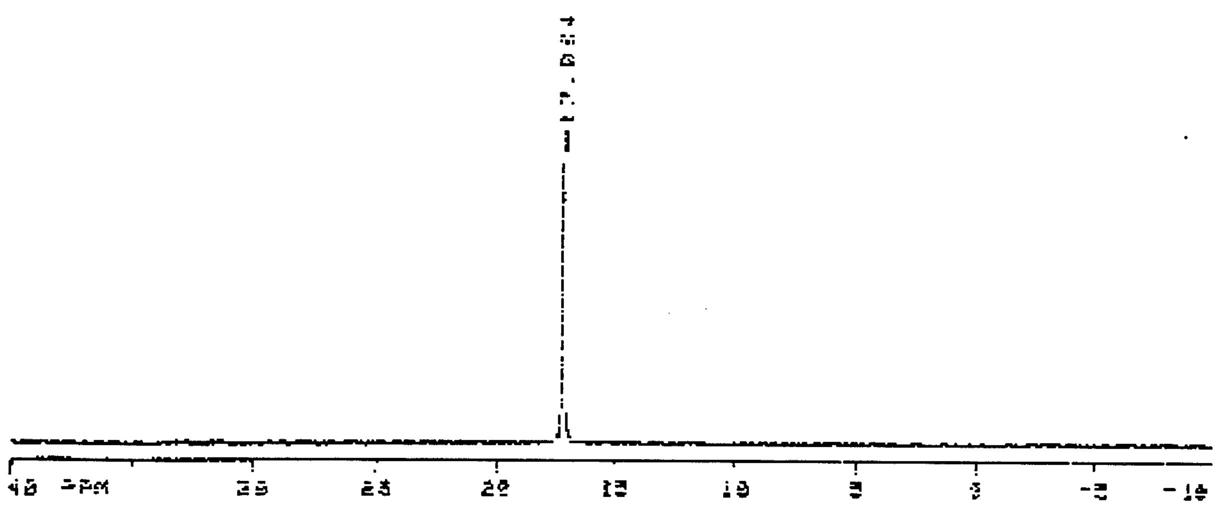

Figure 3. NMR Spectrum of Water Extract of Agent A in Jet A Fuel

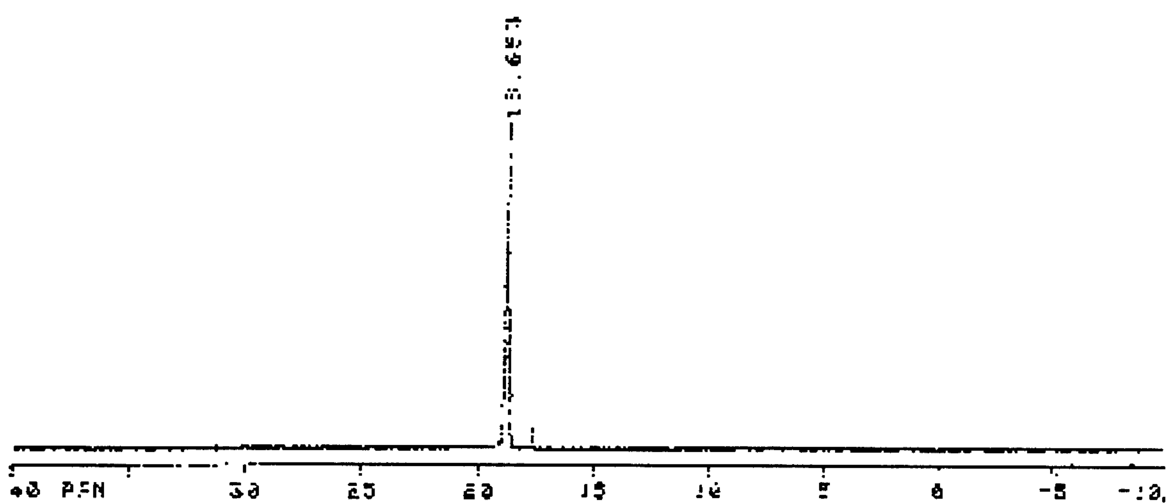

Figure 4. NMR Spectrum of Water Extract of Agent C in Jet A Fuel

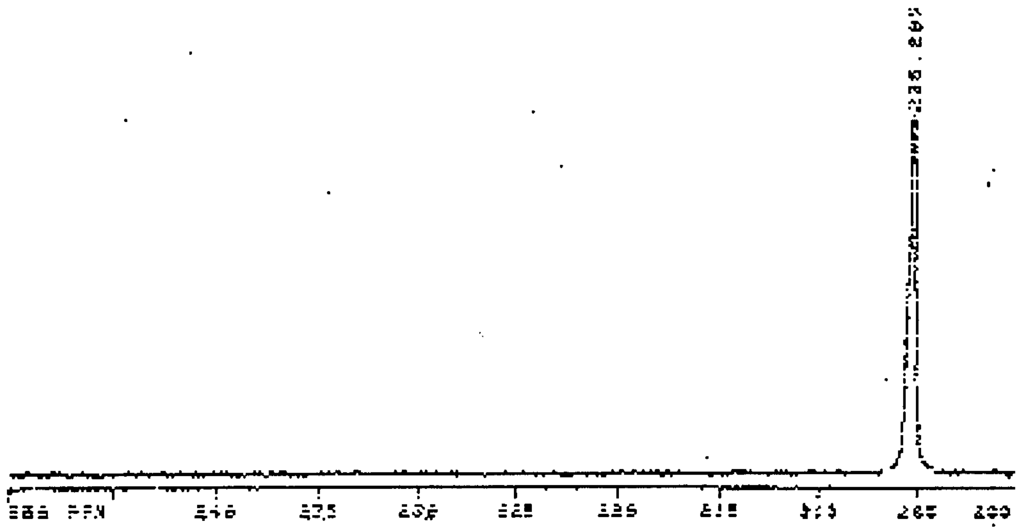

Figure 5. NMR Spectrum of Agent B in Jet A Fuel

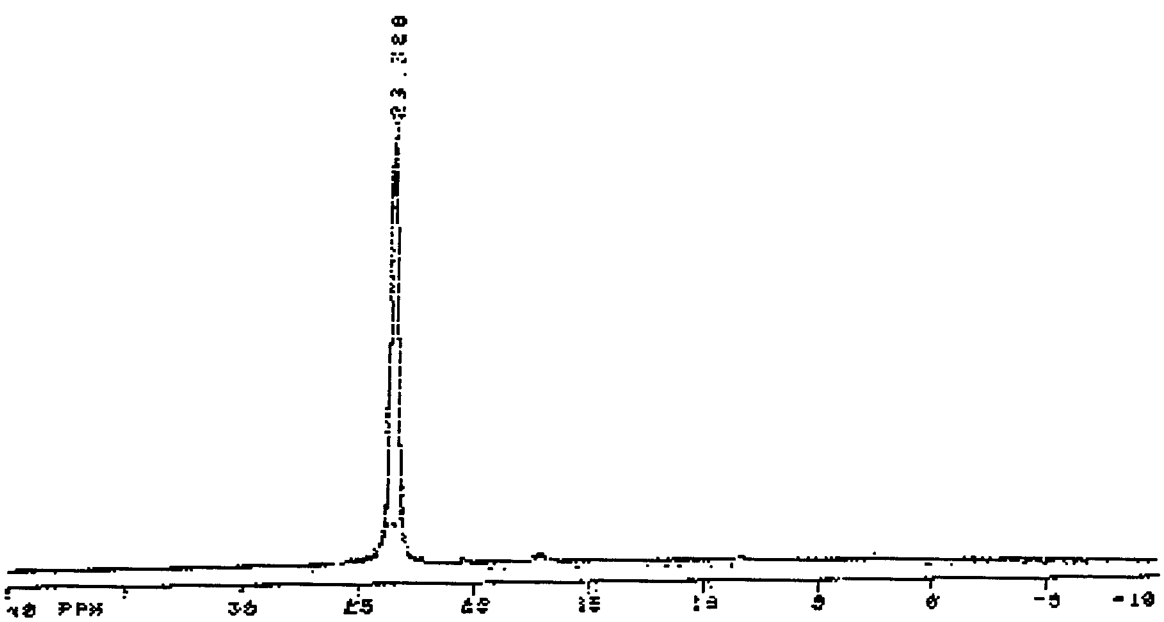

Figure 6. NMR Spectrum of Agent D in Jet A Fuel

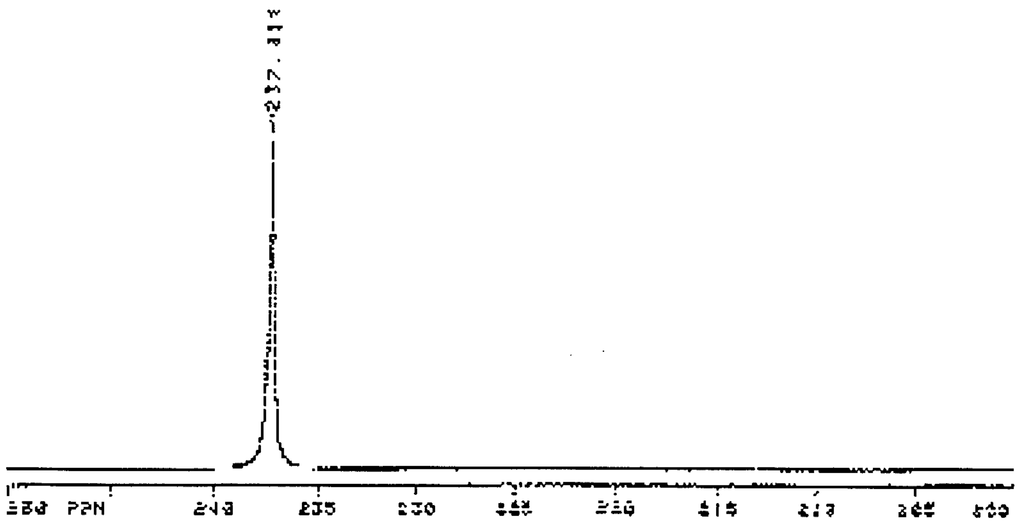

Figure 7. NMR Spectrum of Agent D in Jet A Fuel

Section 3 Fabrication/Standardization

FABRICATION OF DEMONSTRATION MODELS:

A Test Kit for Fuel Acid Contamination (TKFAC) was developed based upon the results of the laboratory investigation. Five Test Kits were fabricated for demonstration and concept feasibility purposes. Three of the Test Kits were shipped to Army units in Saudia Arabia in time for use during Operation Desert Storm. The Test Kit consists of prepackaged tubes containing indicator solution, sampling equipment, safety equipment, instructions and a color comparison chart all packaged in a portable, disposable case.

The indicator solution was based on an aqueous mixture of the following pH indicators:

- Methyl Orange solution – 0.008 percent
- Bromocresol Green solution – 0.002 percent

This concentration allows for a highly visible change of color when acid contaminated fuel (at a minimum concentration of 100 ppm) of any type is contacted by the indicator solution at a 1:1 ratio. This color change is shown on a color comparison chart (color bar) indicating whether the fuel is acceptable ("GO") or unacceptable ("NO-GO"). The color change occurs whenever the indicator solution pH is reduced to 3.5. or lower.

The indicator solution was prepared in a single batch and distributed in 10 mL allotments into sample tubes. The sample tubes were 30 mL disposable plastic (polysulfone) centrifuge tubes with screw closures. The tubes were indelibly marked at the 20 mL level to indicate the quantity of fuel to be added. The procedure called for adding a 10 mL sample of fuel to the tube, agitating (shaking) for ten seconds, then noting the color of the bottom (aqueous) layer and comparing it to the color comparison chart. The tubes are not reusable. The use of the tubes in the field is shown in Figure 8.

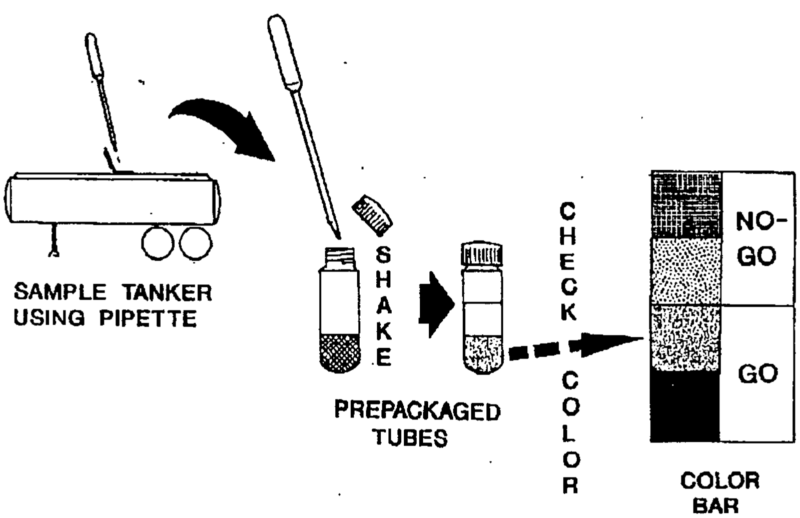

Figure 8. Use of the TKFAC in the Field

Included with the 30 prepackaged tubes were ten plastic disposable sampling pipettes (10 mL capacity), two pairs of plastic disposable safety gloves, two pairs of plastic disposable safety glasses, disposable paper wipers, and a laminated card containing instructions and the color comparison chart. All components are packed in a 40 cm x 20 cm x 20 cm cardboard or plastic 'toolbox' equipped with a hinged lid and handle. The whole Test Kit is considered expendable, i.e., after the tubes are used up the Kit can be thrown out. A sketch of the TKFAC model configuration is shown in Figure 9.

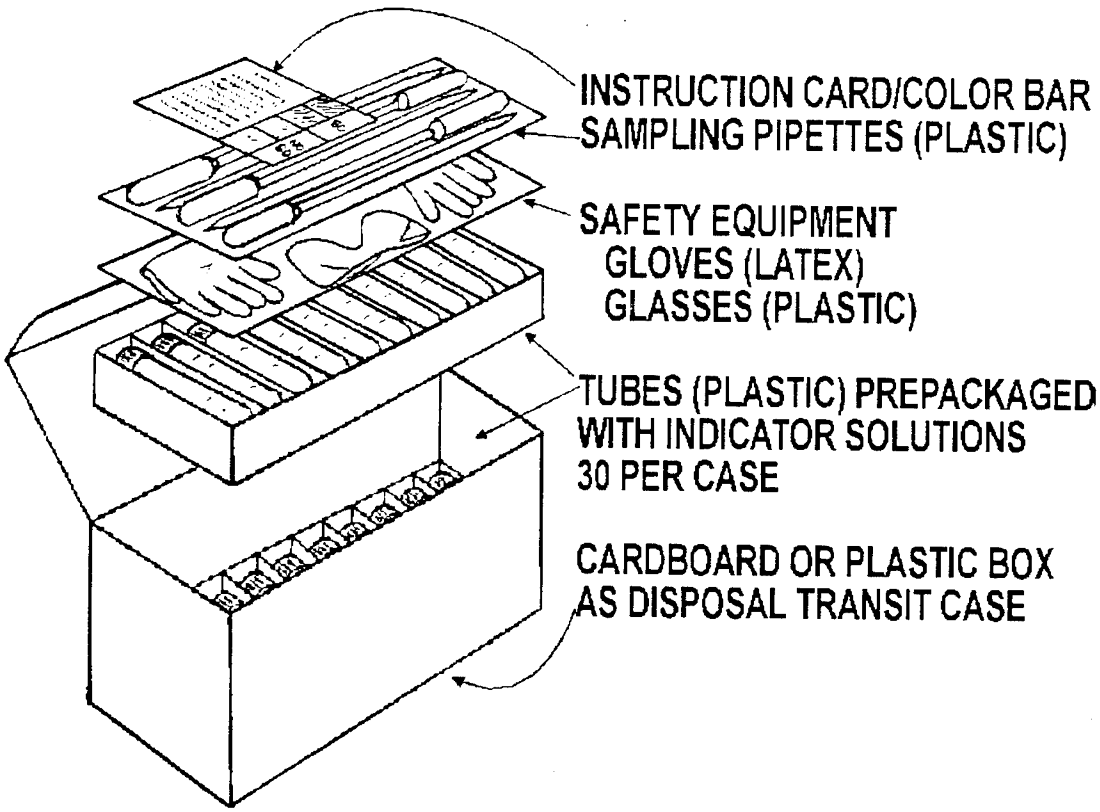

Figure 9. TKFAC Model Configuration

STANDARDIZATION

A purchase description based on this design was prepared to allow or procurement of the Test Kit. A draft of this purchase description is shown as Appendix A.

Section 4 Summary and Conclusions

A field test method for detecting the presence of unique acid contamination in hydrocarbon fuels was developed which is quick and simple and can be applicable to field environments.

An indicating solution containing 0.008 percent Methyl Orange and 0.002 percent Bromocresol Green was found to be an appropriate means of indicating acid contamination in fuel. This solution was found to be stable at extreme temperatures.

A ratio of fuel sample to indicating solution of 1:1 will work satisfactorily.

The concentrations of acid contamination or acid agents could be effected by reaction with fuel components or additives resulting in the precipitation or settlement of the reaction products. However, the process would not lead to false positives during testing.

The reaction products of acid contamination or acid agents are highly water soluble. Water could possible be used an effective decontaminating agent.

Based on the results of this effort, quantity production of the Test Kit for Fuel Acid Contamination (TKFAC) would appear to be highly feasible.

References

1. Memorandum, STRBE-VF (750-51b) to STRBE-Z, 12 October 1990, Subject: Feasibility Program for Field Detection of Anti-Materiel Agents (FAMA).

2. U.S. Military Specification MIL-L-2104E, Lubricating Oil, Internal Combustion Engine, Combat/Tactical Service, 1 August 1988.

3. U.S. Military Specification MIL-T-5624N, Turbine Fuel, Aviation, Grades JP-4, JP-5 and JP-5/JP-8 ST, 10 February 1989.

4. U.S. Military Specification MIL-F-46162D, Fuel Diesel, Referee Grade, 23 November 1992.

5. U.S. Military Specification MIL-T-83133C, Turbine Fuels, Aviation, Kerosene Types, NATO F-34 (JP-8) and NATO F-35, 22 March 1990.

6. U.S. Military Specification MIL-T-85470A, Inhibitor, Icing, Fuel System, High Flash NATO Code Number S-1745, 8 August 1990.

7. Federal Specification VV-F-800D, Fuel Oil, Diesel, 27 October 1987.

8. American Society for Testing and Materials Standard D910, Specification for Aviation Gasolines, 1989.

9. American Society for Testing and Materials Standard D 1655, Specification for Aviation Turbine Fuels, 1989.

10. American Society for Testing and Materials Standard D 4814, Specification for Automotive Spark-Ignition Engine Fuel, 1990.

11. American Society for Testing and Materials Standard E70, Test of Acidity, Alkalinity, or pH of Aqueous Solutions, with Glass Electrode, 1990.

12. American Society for Testing and Materials STP 509A, Single Cylinder Engine Tests for Evaluating the Performance of Crankcase Lubricants, Caterpillar 1H2 Test Method, Appendix F, 1979.

PD ____ ____

DRAFT

PURCHASE DESCRIPTION

TEST KIT FOR FUEL ACID CONTAMINATION

This purchase description is approved for use by the U. S. Army Tank Automotive Research, Development and Engineering Center, Department of the Army.

1. SCOPE

1.1 Scope. This purchase description covers a field portable test kit for determining the presence of a unique acid contaminant in military hydrocarbon fuels.

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications and standards. The following specifications and standards form a part of this purchase description to the extend specified herein. Unless otherwise specified, the issues of these documents shall be those listed in the issue of the Department of Defense Index of Specifications and Standards (DoDISS) and supplement thereto, cited in the solicitation.

SPECIFICATIONS

FEDERAL

L-P-535	-Plastic Sheet (Sheeting); Plastic Strip; Vinyl Chloride Polymer and Vinyl Chloride-Vinyl Acetate Co-Polymer Rigid.
UU-T-595	-Towel, Wiping, Paper, Industrial and Institutional.
PPP-B-601	-Boxes, Wood, Cleated- Plywood.
PPP-B-636	-Boxes, Shipping, Fiberboard.
VV-F-800	-Fuel Oil, Diesel.
A-A-1432	-Towel, Paper (Industrial Wiping).

MILITARY

MIL-G-3056	-Gasoline, Automotive, Combat.
MIL-T-5624	-Turbine Fuel, Aviation, Grades JP-4 and JP-5.
MIL-T-22085	-Tape, Pressure Sensitive, Adhesive, Preservation and Sealing.

MIL-F-46162	-Fuel Oil, Diesel, Referee Grade.
MIL-T-83133	-Turbine Fuel, Aviation, Kerosene Type, Grade JP-8.

STANDARDS

FEDERAL

FED-STD-313 -Material Safety Data, Transportation Data and Disposal Data for Hazardous Materials Furnished to Government Activities.

MILITARY

MIL-STD-105	-Sampling Procedures and Tables for Inspection by Attributes.
MIL-STD-129	-Marking for Shipment and Storage.
MIL-STD-130	-Identification Marking of U.S. Military Property.
MIL-STD-810	-Environmental Test Methods.
MIL-STD-889	-Dissimilar Metals.
MIL-STD-1472	-Human Engineering Design Criteria for Military Systems, Equipment and Facilities.

(Copies of specifications and standards required by contractors in connection with specified acquisition functions should be obtained from the contracting activity or as directed by the contracting officer.)

2.2 Other publications. The following document(s) form a part of this purchase description to the extend specified herein. The issues of the documents which are indicated as DoD adopted shall be the issue listed in the current DoDISS and the supplement thereto, if applicable.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

D 910	-Standard Specification for Aviation Gasolines.
D 975	-Standard Specification for Diesel Fuel Oils.
D 1655	-Standard Specification for Aviation Turbine Fuels.
D 3953	-Strapping, Flat Steel and Seals.
D 4675	-Selection and Use of Flat Strapping Materials.
D 4814	-Standard Specification for Automotive Spark-ignition Engine Fuel.
E 70	-Test of Acidity, Alkalinity, or pH of Aqueous Solutions, with Glass Electrode.

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103).

(Non-Government standards and other publications are normally available from the organizations which prepare or which distribute the documents. These documents also may be available in or through libraries or other informational services.)

2.4 Order of precedence. In the event of a conflict between the text of this purchase description and the reference cited herein, the text of this purchase description shall take precedence. Nothing in this purchase description, however, shall supersede applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Description. The Test Kit for Fuel Acid Contamination (hereafter called Test Kit) is a compact, portable kit intended for use by field solders, fuels handling personnel, or tank truck drivers in order to determine the presence of a unique acid contaminant in military and commercial hydrocarbon fuels. The test is a qualitative method only, applicable to field use by essentially untrained personnel. Test results should indicate either acceptable ("GO") or unacceptable ("NO-GO") with a surety factor sufficient to eliminate the possibility of false negatives and minimize the possibilities of false positives. Acid determination will be by means of standard indicator solutions prepackaged in sealed tubes. Other components of the Test Kit will be sampling equipment, personnel protective equipment, paper wipers and a plastic laminated card imprinted with a color chart as well as general and safety instructions. All components will be packaged in a transit case. Case and components must be sufficiently durable to withstand the rough handling common to a military support function.

3.2 First article. Unless otherwise specified (see 6.2), a sample shall be subjected to first article inspection (see 4.3 and 6.3). Any changes or deviations of Test Kits from the approved first article during production will be subjected to the approval of the contracting officer. Approval of the first article will not relieve the contractor of his obligation to furnish Test Kits conforming to this Purchase Description.

3.3 Materials. Materials in contact with hydrocarbon fuels should be fuel compatible and resist deterioration. Materials in continuous contact with indicator solutions (see 3.5) should be fully compatible with the solutions and resist deterioration. All materials should be noncombustible, fire-resistant or self-extinguishing.

3.3.1 Plastics. Plastic materials shall be resistant to discoloration or attack by hydrocarbon fuels or indicator solutions (see 3.5), where applicable, and should resist becoming crazed or opaque under exposure to sunlight.

3.3.2 Metals. Metals shall be corrosion resistant.

3.3.2.1 Materials deterioration prevention and control. The Test Kit shall be fabricated from compatible materials, inherently corrosion resistant or treated to provide protection against the various forms of corrosion and deterioration that may be encountered in any of the applicable operating and storage environments to which the Test Kit may be exposed.

3.3.2.2 Dissimilar metals. Dissimilar metals shall not be used in intimate contact with each other unless protected against galvanic corrosion. Dissimilar metals and methods of protection are defined and detailed in MIL-STD-889.

3.3.2.3 Identification of materials and finishes. The contractor shall identify the specific material, material finish or treatment for use with component and subcomponent, and shall make information available upon request to the contracting officer or designated representative.

3.3.3 Recovered materials. For the purpose of this requirement, recovered materials are those materials which have been collected from solid waste and reprocessed to become a source of raw materials as distinguished from virgin raw materials. The components, pieces, and parts incorporated in the Test Kit may be newly fabricated from recovered materials to the maximum extent practical, provided the Test Kit meets all the other requirements of this purchase description. Used, rebuilt, or remanufactured components, pieces, and parts shall not be incorporated in the Test Kit.

3.4 Performance characteristics.

3.4.1 Applicable fuels. The Test Kit shall be usable with military and commercial hydrocarbon fuels as follows:

VV-F-800	-Fuel Oil, Diesel.
MIL-G-3056	-Gasoline, Automotive, Combat.
MIL-T-5624	-Turbine Fuel, Aviation, Grades JP-4 and JP-5.
MIL-T-83133	-Turbine Fuel, Aviation, Kerosene Type, Grade JP-8.
ASTM D 910	-Aviation Gasoline.
ASTM D 975	-Diesel Fuel Oils.
ASTM D 1655	-Aviation Turbine Fuels.
ASTM D 4814	-Automotive Spark-ignition Engine Fuel.

3.4.2 Sample size. Size of fuel sample necessary to make one determination (presence of acid contamination) shall be large enough to be representative and to be highly visible in the sample tube (see 3.5) but should not exceed 10 mL (0.33 fl oz).

3.4.3 Indicator solution. Detection of the acid contaminant in fuel shall be by contact with aqueous solutions of indicator solutions. Indicator solutions must give a highly visible color change for a pH change from 4.0 to 3.5. An aqueous indicator solution with a pH of 4.0 must show LITTLE OR NO CHANGE. An aqueous indicator solution with a pH of 3.5 must show COMPLETE CHANGE. The color change must be visible to the unaided eye even in subdued light. A combination of indicator solutions that has been found to work satisfactorily consists of the following:

Methyl orange solution - 0.008 percent
Bromocresol green solution - 0.002 percent

Such a solution will indicate the following colors: pH 4.0 - green or blue-green; pH 3.5 - yellow or yellow-orange.

Indicator solution shall be prepackaged in sample tubes (see 3.5) at a fixed capacity not to exceed that for the fuel sample size, i.e., 10 mL (.33 fl oz), at a tolerance of + 5 %.

3.5 Sample tubes. Sample tubes must be fabricated of transparent unbreakable plastic (polysulfone or equal) compatible with the indicator solution and with all hydrocarbon fuels. Tubes may be flat or round bottom (centrifuge tubes) and should have a screw closure capable of providing a positive seal to prevent leakage of liquids in all positions and at an altitude of 4750 meters (15,000 ft) as set forth in Method 500.2 of MIL-STD-810. Capacity of tubes should be no greater than 30 mL (1 fl oz.) to allow for the prepackaged indicator solution (see 3.4.3), fuel sample (see 3.4.2) and space above for shaking or agitation. Tubes shall be permanently marked to indicate the fuel sample fill level (with the indicator solution already in). Marking must be indelible, water and fuel resistant.

3.6 Sampling pipettes. Sampling pipettes shall be of one-piece disposable plastic type equipped with a squeeze bulb. Capacity shall be sufficient to allow for transfer of one fuel sample (10 mL or 0.33 fl oz, minimum). Total length should be 30 + 2 cm (11.8 + 1 in) to allow for manual sampling from the hatchway of tank vehicles and for storage within the Transit Case (see 3.11).

3.7 Sampling beakers. Sampling beakers should be of the disposable plastic type with a pouring spout. Capacity shall be nominal 50 mL (1.65 fl oz) with capacity indication marked on the side.

3.8 Safety gloves. Gloves will be plastic disposable, resistant to hydrocarbons and acids.

3.9 Safety glasses. Glasses will be plastic disposable, resistant to hydrocarbons and acids. Lenses will be clear. Glasses must provide wearer protection against splashes from acid contaminated fuel.

3.10 Paper towels. Thirty (30) sheets of industrial grade disposable paper toweling meeting the requirements of A-A-1432, UU-T-595, or commercial equivalent will be supplied for cleaning.

3.11 Plastic laminated card. A plastic laminated card will show a color comparison chart to indicate to the operator the expected color changes in the indicator solutions. Color chart must show actual color changes (photographic or facsimile) in the indicating solution for neat (unadulterated) fuel (pH 4.0 or greater) and for acid contaminated (adulterated) fuel (pH 3.5 or less). Colors must agree with those encountered in 3.4.3 and 4.5.2.1.2. Titles with the color chart will indicate "GO" or acceptable fuel (i.e., non acid contaminated) and "NO-GO" or unacceptable fuel (i.e., acid contaminated). Included on the card will be general instructions (condensed from the manual) and special safety warnings. Card shall be 0.63 to 0.76 mm (0.025 to 0.030 in.) thick with an opaque white background. Card must be legible in subdued light (see 3.19 and 4.5.2.6). Card shall be fuel and water resistant, laminated on both sides with material specified in L-P-535, composition A, Type II. Dimensions of the card shall not be greater that the two largest dimensions of the Transit Case (see 3.12).

3.12 Transit case. The transit case will be fabricated of rigid or semi-rigid plastic or elastomeric material. It shall be fuel resistant and non-absorbent. All materials used in the case should be fire resistant or self extinguishing and resistant to microbiological deterioration, to the greatest extent possible. It will come with a hinged top and a carrying handle. It may contain one or more interior trays to facilitate loading and packing. All components should fit in the case compactly so as to maximize the space available and minimize the exterior dimensions of the case. Case packed with its contents should meet the transportability requirements of 4.5.2.3. The outside of the case shall be colored forest green.

3.13 Installation of components in transit case. All components (see 3.5 through 3.11) should be installed in the transit case (see 3.12) so as to minimize the chance of damage, optimize the space available and provide ready access for the operator.

3.14 Shelf life. The Test Kit will be designed for a minimum shelf life of five (5) years.

3.15 Environmental requirements. Test Kit will be designed to operate under adverse weather conditions to include rain, dust, humidity, salt for, solar radiation as well as temperature extremes.

3.15.1 Operating climatic conditions. The Test Kit shall be capable of being operated in any temperature above the freezing point of water, 0°C (32°F), to 49°C (120°F) (climatic design Basic Hot and Basic Cold per MIL-STD-810 adjusted to account for the freezing point of the indicator solutions) when tested in accordance with 4.5.2.2.1

3.15.2 Storage climatic conditions. The Test Kit shall not be damaged in any ambient temperature from 63°C (145°F) to minus 33°C (minus 27°F) (climatic design type Basic Hot and Basic Cold per MIL-STD-810) when tested in accordance with 4.5.2.2.2

3.16 Transportability. The Test Kit shall be capable of withstanding the vibrational stress and shock encountered by all modes of transportation,including tracked combat vehicles, when tested in accordance with 4.5.2.3.

3.17 Marking. Each Test Kit will be marked in accordance with MIL-STD-130. Marking will be in a conspicuous location on the exterior of the Transit Case. Marking will consist of the following:

TEST KIT FOR FUEL ACID CONTAMINATION
97403-[TOP ASSEMBLY DRAWING NUMBER]
MFR- [MFR NAME OPTIONAL]
NSN:

3.18 Safety. The Test Kit shall be designed using sound safety and human engineering practices and will have no uncontrolled safety and health hazards associated with its use. Safety warnings, particularly regarding the avoidance of acid contaminated fluids on the skin or eyes should be placed in a conspicuous place. Proper safety equipment to include glasses and gloves shall be included within each Test Kit. Material Safety Data Sheets shall be printed in accordance with FED-STD-313 and one sheet will be included within each Test Kit.

3.19 Human factors. The Test Kit shall be designed in accordance with accepted criteria of design for Human Factors as described in MIL-STD-1472. The Test Kit shall be operable during day and night by 5th through 95th percentile soldiers in combat environments. Particular design attention shall be given, but not limited to MIL-STD-1472: 4 (General Requirements), 5.5 (Labeling), 5.6 (Anthropometry), and 5.13 (Hazards and Safety).

3.20 Operation under adverse conditions. The Test Kit must be fully operable by personnel wearing MOPP IV protective clothing and arctic mittens, and under reduced illumination.

3.21 Workmanship. Test Kit shall be fabricated free from any defects which could affect surety or durability. The plastic used in components and in the Transit Case must be free of cracks, stress patterns, discolorations, opacity, and crazing. Sampling tubes and sampling beakers must be leakproof when filled with fluid. All markings, labels and printed matter on the Transit Case or laminated card shall be legible and protected from environmental damage.

3.22 Surety. The surety of the Test Kit shall be expressed as the probability of indicating aqueous solutions of pH 4.0 as being "acceptable" and of aqueous solutions of pH 3.5 as being "unacceptable" (see 3.4.3 and 4.5.2.1). A probability of 0.95 at a 90 percent confidence level constitutes the surety requirement. No requirement for reliability exists as a failure will not trigger a maintenance action. No requirement for maintainability exists as any Test Kit not functioning properly shall be replaced in the field. Therefore, operational availability cannot be addressed.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements specified herein. Except as otherwise specified in the contract or purchase order, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The government reserves the right to perform any of the inspections set forth in this purchase description where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.1.1 Responsibility for compliance. All items must meet all requirements of section 3 and 5. the inspection set forth in this purchase description shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the purchase description shall not relieve the contractor of the responsibility of assuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling in quality conformance does not authorize submission of known defective material, either indicated or actual, or does it commit the Government to acceptance of defective material.

4.2 Classification of inspections. The inspection requirements specified herein are classified as follows:

a. First article inspection (see 4.3).

b. Quality conformance inspection (see 4.4).

c. Inspection of packaging (see 4.6).

4.3 First article testing.

4.3.1 First article examination. The ten first articles shall be examined as shown in 4.5.1 (Table I) in the order shown. The presence of one or more defects shall be cause for rejection.

4.3.2 First article tests. The first ten articles shall be tested as shown in 4.5.2 (Table II) in the order shown. Failure of any test shall be cause for rejection.

4.4 Quality conformance inspection.

4.4.1 Quality conformance examination. Each Test Kit shall be examined as shown in 4.5.1 (Table I) in the order shown. Inspection shall be Normal Inspection Level (I). The presence of one or more defects shall be cause for rejection.

4.4.2 Quality conformance tests. Sample in conformance with MIL-STD-105, Normal Inspection Level (I). Tests shown in 4.5.2 (Table II) shall be performed in the order shown. The presence of one or more defects shall be cause for rejection.

4.5 Inspection schedule.

4.5.1 Examination.

Table I Examination schedule

First Article	Quality Conformance	Defect	Requirement Paragraph
X	X	101. Materials not as specified.	3.3
X	X	102. Materials not resistant to corrosion and deterioration for the applicable storage and operating environment.	3.3.2.1
X	X	103. Dissimilar metals not in accordance with MIL-STD-889.	3.3.2.2
X	X	104. Materials and material finish or treatment information not available.	3.3.2.3
X	X	105. Indicator solutions missing or not as specified.	3.4.3
X	X	106. Sample tubes missing or not as specified.	3.5
X	X	107. Sampling pipettes missing or not as specified.	3.6

X	X	108. Sampling beakers missing or not as specified.	3.7
X	X	109. Safety gloves missing or not as specified.	3.8
X	X	110. Safety glasses missing or not as specified.	3.9
X	X	111. Paper towels missing or not as specified.	3.10
X	X	112. Laminated card missing or missing or not as specified.	3.11
X	X	113. Transit case not as specified.	3.12
X	X	114. Components not installed as specified.	3.13
X	X	115. Marking not as specified.	3.17
X	X	116. Material Safety Data Sheet missing.	3.18
X	X	117. Workmanship not as specified.	3.21

4.5.2 Tests.

Table II Test schedule.

First Article	Quality Conformance	Test	Test Paragraph	Requirement Paragraph
X	-	Acid indication	4.5.2.1	3.4.3
X	-	Environmental	4.5.2.2	3.15
X	-	Transportability	4.5.2.3	3.16
X	-	Human factors	4.5.2.4	3.19
X	-	Operation under adverse conditions	4.5.2.5	3.20
X	-	Reliability, availability, and maintainability.	4.5.2.6	3.22

4.5.2.1 Acid indication.

4.5.2.1.1 False positives. Obtain quantities of Fuel Oil, Diesel, Referee Grade (MIL-F-46162) and Turbine Fuel (MIL-T-83133). Add an appropriate sample size (see 3.4.2) of each fuel separately to each sample tube in each Test Kit, reseal cap and agitate for ten (10) seconds. After settling, there should be LITTLE OR NO CHANGE in the color of the indicator solution in the bottom of the tube.

4.5.2.1.2 pH test. Each sample tube in each Test Kit shall be tested. Prepare titration apparatus and pH electrode as described in ASTM E 70. Range of pH meter readout should include the range of 3.0 to 4.5. Pour contents of sample tube into transparent beaker and insert pH electrode. Titrate dilute (0.1 N) hydrochloric acid (HCl) into the indicating solution until a pH of 4.0 + 0.1 is indicated. Stop titration and note color of solute. There should be LITTLE OR NO CHANGE. Continue titration into indicating solution until a pH of 3.5 + 0.1 is indicated. Stop titration and note color of solute. There should be COMPLETE CHANGE of color. Color change must be highly visible to comply with requirements of 3.4.2. Failure to meet the pH and color change requirements shall constitute failure of this test.

4.5.2.2 Environmental.

4.5.2.2.1 Operational. Detection tests (see 4.5.2.1) shall be repeated at both low and high temperatures.

4.5.2.2.1.1 Low temperature. Indicator solutions and test fuels, where applicable, (see 4.5.2.1.1) shall be subjected to an environmental temperature of minus 33 + 1°C (minus 28 + 2°F) for a period of 24 hours. The indicator solutions will then be allowed to thaw with the temperatures of both fluids brought to 0 + 1°C (32 + 2°F) for a minimum of two hours prior to testing. Temperature will be maintained at this same level during testing. Failure to obtain results detailed in 4.5.2.1 shall constitute failure of this test.

4.5.2.2.1.2 High temperature. Indicator solutions and test fuels, where applicable, (see 4.5.2.1.1) shall be subjected to an environmental temperature of 63 + 1°C (145 + 2°F) for a period of 24 hours. Temperature for both fluids shall be lowered to 49 + 1°C (120 + 2°F) for a minimum of two hours prior to testing. Temperature will be maintained at this level during testing. Failure to obtain results detailed in 4.5.2.1 shall constitute failure of this test.

4.5.2.2.2 Storage. Each Test Kit shall be tested as specified herein.

4.5.2.2.2.1 Low temperature. The Test Kit shall be subjected to a low temperature environment of minus 33 + 1°C (minus 38 + 2°F) for a period of 24 hours and tested in accordance with MIL-STD-810, method 501.2, procedure I-Storage.

4.5.2.2.2.2 High temperature. The Test Kit shall be subjected to a high temperature environment of 63 + 1°C (145 + 2°F) for a period of 4 hours after an initial soak of 49 + 1°C (120 + 2°F), raising and lowering of temperature to be accomplished within a time period of one hour for a total cycle of 12 hours. Except for the minimum total hours (84 hours), complete testing in accordance with MIL-STD-810, method 501.2, procedure I-Storage.

4.5.2.3 Transportability. Each Test Kit shall be tested for vibration in accordance with the Loose Cargo Transport Category for Equipment carried on ground vehicles as unrestrained cargo, cited under method 514.4 of MIL-STD-810. Test time should be 3 hours with only one item on the test bed at a time. Each Test Kit shall be tested for shock in accordance with the procedure covering transit drop under method 516.4 of MIL-STD-810, height of drop to be 122 cm (48 in.). Upon completion of tests, each Test Kit will be examined for damage. Any evidence of permanent deformation, cracks, broken components or leaks in the prepackaged tubes shall constitute failure of this test.

4.5.2.4 Human factors. Each Test Kit shall be evaluated throughout the test cycle to determine compliance with the requirements of 3.19. Nonconformance to 3.19 shall constitute failure of this test.

4.5.2.5 Operation under adverse conditions. Each Test Kit shall be tested at least once under simulated adverse conditions. Test should include simulated field use of the Test Kit and the and the detection of color changes. All tests are to be performed with the operator wearing a real or simulated MOPP IV face mask. In addition, the simulated field test (see 4.5.2.6.1) shall be performed wearing real or simulated arctic mittens. Illumination for both tests shall be limited to that provided from a single 3VDC battery operated hand held flashlight with a lens diameter not exceeding 5 cm (2 in.).

4.5.2.5.1 Simulated field test. Obtain 100 mL or more of any of the military fuels listed in 4.5.2.1.1 and place in a beaker. Put on Army arctic mittens. Open the Test Kit and use one of the sampling pipettes to extract the proper size fuel sample from the beaker. Select one of the prepackaged sample tubes and empty pipette sample into it. Shake tube for ten (10) seconds and let settle. Note lower layer. There should be LITTLE OR NO CHANGE in the color of the indicator solution. Inability to perform this test or the a false positive indication shall constitute failure of this test.

4.5.2.5.2 Acid indication test. Perform the pH titration test as described in 4.5.2.1.2 on one randomly selected prepackaged sample tubes. Failure to obtain results detailed in 4.5.2.1.2 shall constitute failure of this test.

4.5.2.6 Surety. Each Test Kit shall be evaluated throughout the test cycle to determine compliance with the requirements of 3.22 Nonconformance with this requirement shall constitute failure of this test.

4.6 Inspection of Packaging.

4.6.1 Quality conformance inspection of packaging.

4.6.1.1 Unit of product. For the purpose of inspection, the unit of product shall be a completed pack of a single Test Kit prepared for shipment.

4.6.1.2 Sampling. Sampling for examination shall be in accordance with MIL-STD-105.

4.6.1.3 Examination. Samples selected in accordance with 4.6.1.2 shall be examined for the following major defects. Presence of one or more defects shall the cause for rejection.

115. Test Kit not preserved as specified (see 5.1).

116. Six complete Test Kits not packed in a shipping box as specified for Level A (see 5.2.1 and 5.2.2).

117. Strapping not as specified (see 5.2.1 and 5.2.2).

118. Marking illegible, incomplete, incorrect, or missing (see 5.3).

5. PACKAGING

5.1 Preservation. Preservation shall be Level A.

5.1.1 Level A. Each Test Kit, defined as the Transit Case filled with the necessary components, shall be placed in a snug fitting fiberboard box conforming to PPP-B-636, minimum grade W5c, style FPF (five panel folder). Box closure shall be as specified in the appendix to the box specification.

5.2 Packing. Packing shall be level A.

5.2.1 Level A. Six (6) complete Test Kits, preserved as specified in 5.1, shall be packed together in a close fitting box conforming to PPP-B-601, overseas type, style I. Box closure shall be in accordance with the appendix to the box specification. Strapping shall be in accordance with ASTM D 3953, type 1 or 2, zinc-coated, size as applicable, and ASTM D 4675.

5.3 Marking. In addition to any special or identification markings required by the contract or purchase order (see 6.2), each container shall be marked in accordance with MIL-STD-129.

6. NOTES

6.1 Intended use. the Test Kit for Fuel Acid Contamination is intended for use by military petroleum handling personnel and by field soldiers to determine the presence of special acid contamination in bulk hydrocarbon fuel supplies.

6.2 Ordering data. Procurement documents should specify the following:

a. Title, number, and date of this Purchase Description.

b. When a first article is not required (see 3.2).

c. Time frame required for submission of first articles.

d. Special marking required (see 5.3).

6.3 First articles. First articles shall be ten (10) preproduction models. Arrangements should be made with the contracting officer prior to initiation of first article examination and tests to arrange for monitoring. If first article examination and test results are approved, the first articles can become part of the production lot (see 3.2). Consumable fluids and components used as part of the test may be replaced in the preproduction models.

6.4 Material Safety Data Sheets. Contracting officer will identify those activities requiring copies of completed Material Safety Data Sheets prepared in accordance with FED-STD-313. The pertinent Government mailing addresses for submission of data are listed in FED-STD-313.

6.5 Subject term (key word) listing.

Fuel testing
Fuel test kits
Anti-materiel agents
Field testing
Acidic contaminant

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